

On-Demand Ultratrace Vapor Calibration and Testing Device Enabled by Piezoelectric Technology

Innovative chemical sensors are emerging to detect the emanating vapor plumes of concealed high explosives and toxic chemicals, the concentrations of which may be present only in the parts-per-trillion range. Unfortunately, no reliable mechanism exists for calibrating these devices, or for testing/intercomparing performance under appropriate conditions. The sensor industry therefore lacks the measurement capability to pursue tangible detection goals, and without performance verification, the technology is unacceptable to user groups such as the Transportation Security Administration (TSA). NIST has engaged with this community to address these issues.

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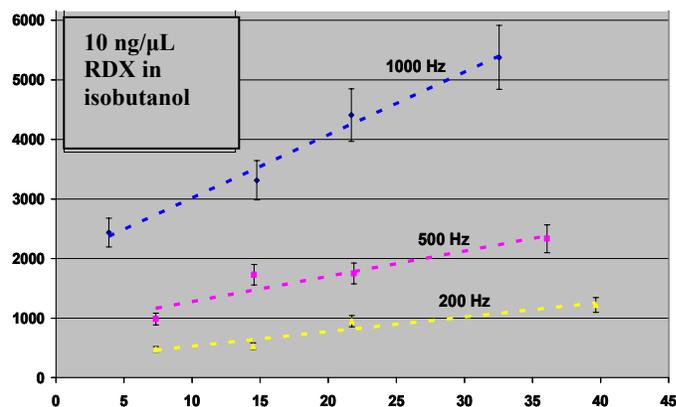
NIST designed a piezoelectric device and had the prototype built to specifications by MicroFab Technologies. The photograph shows the fabricated Micro-droplet Vapor Generator (MVG), with six solution reservoirs feeding into a bank of piezoelectric nozzles. The nozzles dispense droplets onto the heated white ceramic tube seen within the cooled flanged body. Vaporized droplets are entrained in an air stream entering behind the device. Similar to an inkjet printer, the MVG can dispense, in a drop-on-demand, pulsed, or continuous fashion, precise numbers of standardized solution droplets at controlled jetting rates onto a heated surface. At optimized temperatures, the droplets vaporize and blend into a calibrated air stream to generate reproducible analyte/gas mixing ratios across seven orders of magnitude, the ratio being dependent upon the number of nozzles employed, the solution concentration, the jetting rate, and the air flow.



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This year, we built and tested the MVG with trace solutions of RDX, PETN, and TNT dissolved in isobutanol ($T_b = 108\text{ }^\circ\text{C}$), and overcame irreproducibility issues resulting from incomplete vaporization of droplets at the heated surface. An optical microscope with strobed illumination was used to monitor the droplet jetting rate and vaporization process. We observed droplet bounce at temperatures between $20\text{ }^\circ\text{C}$ to $120\text{ }^\circ\text{C}$, due to insufficient heat transfer and semielastic collisions on the heated surface, and at temperatures greater than $140\text{ }^\circ\text{C}$, which arose from elastic

collisions with a vapor barrier generated in the film boiling regime. Only temperatures between $120\text{ }^\circ\text{C}$ and $140\text{ }^\circ\text{C}$ allowed the droplets to vaporize completely. MVG capability was then demonstrated by jetting, at various rates, trace solutions of the explosives RDX, TNT, and PETN and measuring the generated vapors by ion mobility spectrometry (IMS). *All analytes behaved similarly; IMS data for RDX are illustrated in the figure in which the IMS response (ion intensity, a.u.) is plotted vs time (s).*



The graph shows the ion mobility response to trace RDX vapors generated by the MVG. The response is nearly linear with droplet jetting rate and sampling time.

The NIST-designed piezoelectric device will enable the reliable on-demand calibration of trace explosive vapor detectors for many compounds in the picogram per gram range (ppt), and offer an acceptable mechanism for performance verification of emerging sensor technologies.

Future Plans: We are passivating interior surfaces to test and minimize wall effects to enabling the quantitative pulsed delivery of trace substances such as plastic explosives that have very low vapor pressures. Work is underway to verify the entire dynamic range and linearity of the MVG device by developing sampling schemes for the generated vapors and isotope dilution mass spectrometry methods for establishing NIST traceability for the measurements. The MVG will also be used to test the vapor collection efficiency of filters and other materials used in walk-through portals that are now being deployed by the TSA at airports and other security checkpoints.